Poster

Combined application of 3D electron diffraction analysis and mechanochemical synthesis for the investigation of novel nanocrystalline reticular materials

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Mechanochemistry finds application in the synthesis of new organic and metal-organic materials such as molecular crystals, salts, cocrystals and polymeric structures. This method allows the formation of products, polymorphs and topologies different from those obtained in solution. However, the structural characterisation of the obtained products is one of the main challenges related to this synthetic approach, due to the presence of nanometric crystals that cannot be analysed by conventional single-crystal X-ray diffraction. Furthermore, powder diffraction methods can be extremely difficult to apply in the case of large unit cells, low symmetry and polyphasic systems. Electron crystallography, especially 3D electron diffraction (3D ED), is an emerging characterisation technique that, due to the most recent developments, can be successfully employed to obtain the structure of nanocrystalline species. This has allowed the structural characterisation of different types of materials, spanning from porous MOFs, COFs and zeolites, to pharmaceutical
APIs [1, 3]. The 3D electron diffraction analysis is usually performed in transmission electron microscopes (TEMs); however, the growing interest in 3D ED also prompted the development of dedicated electron diffractometers. In this contribution, we present a combined approach based on mechanochemistry and 3D ED analysis for the synthesis and structural characterisation of new reticular compounds. In particular, we studied a tetrahedral pyridyl-based SOF [4, 5], zinc(II) and 2,6-pyridine dicarboxylic acid coordination polymers (CPs) [6] and MOFs based on copper(II) and phenolic ligands [7]. The single-crystal 3D ED analyses of metal-organic compounds were performed in a TEM, whereas one of the SOFs was characterised in a new electron diffractometer. The comparison between 3D ED and X-ray powder diffraction data is also discussed.

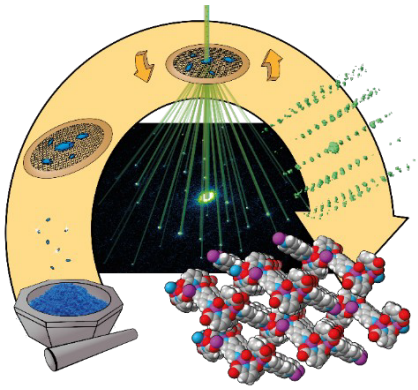

Figure 1. Schematic view of the combined use of mechanochemistry and 3D electron crystallography.

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